FULL SIZE OF Figure 1
IS IN FILE A- 548

FIG. 1

Fig 1

FULL SIZE OF Figure 2
IS IN FILE A- 548

FIG. 2

Fig 3

FULL SIZE OF Figure 3
IS IN FILE A-548

FIG 3

FULL SIZE OF Figure 4
IS IN FILE A- 548

FIG 4

FULL SIZE OF Figure 5
IS IN FILE A- 548

FULL SIZE OF Figure 6
IS IN FILE A- 548

FIG 6

1064

A06913

A0 6913

1 f

Lab. No. MSL-74-133
Req. No. 52467
Date 11-27-73

DOW CHEMICAL U.S.A.
Rocky Flats Division
Mass Spec Laboratory

ANALYSIS REPORT

To R. E. Nelson Bldg. 771 Charge 1371

cc: File Dept.

Ext.

Sample Description:

Sludge from Arco shipping drum.

Identify.

Results of Analyses or Tests:

Infrared, mass spectral, and thermal analytical results indicated the sludge was composed of minor amounts of organic residue, major amounts of inorganic and water.

IR - Inorganic residue as: ammonium ion (NH_4^+)
water
siliceous material (SiO_2)
metal oxides, halides (as PuO_2, CaF_2, etc.)
possible graphitic material?

mass spec outgas - major: water
minor: HCl, HF, hydrocarbon, CO_2, ammonia
trace: dioctylphthalate (bag plasticizer?), POE(polyoxyethylene surfactant?)

TGA-DTA - The only thermal event recorded was a large water volatilization endotherm peaking at 100°C. The thermogravimetric trace indicated the loss of water from 25 to 180°C (equivalent to 37 wt. %), and gradual loss of residual water and other volatiles (equivalent to 27 wt. %) from 180°C to 770°C.

Analysis By MLR, DEA, JRG, RSC, DIH, KJG

File or Plate No. IR 927

Reported By K. J. Grossaint
K. J. Grossaint

Approved By J R Turbett
J. R. Turbett

1940

AQ 7273 2

Rockwell International

Atomics International Division
Rocky Flats Plant
P.O. Box 464
Golden, Colorado 80401

ANALYTICAL REPORT

Organic Mass Spec Lab A07273UINF

To	Account No.	Date	Lab. No.
C. E. Wickland K. Terada Pile J Holt 9.24.75	389	12-1-75	MSL-76-188

Reported by D. I. Hunter K. J. Grossaint
D. I. Hunter and K. J. Grossaint

Approved J R Turbett
J. R. Turbett

Sample Description

Fourteen drums of laundry sludge returned to Rocky Flats from Idaho storage area.

Analysis Results

These drums of laundry sludge were returned to Rocky Flats after a drum of the sludge was accidently punctured and an audible pressure release was noted. Further examination revealed bulged tops on some of these drums.

A special punch fixture was manufactured and used at Rocky Flats to determine the drum pressures and obtain a gas sample for Mass Spectrometric analysis.

Drums no. 17-03909 and 17-03932 had the metal seal broken when received. The most severely deformed drum was 17-03919. A picture of this drum is included in this report.

All 14 drums were marked content code 374 (blacktop, concrete, dirt and sand). Drum 17-03919 was opened and found to contain oil dry around the 55 gallon polyethylene bag of material. These bags were not opened and examined. No free liquid was observed.

The source of pressure in all 14 drums was methane (CH_4) and carbon monoxide (CO). Details appear in the following table.

Drum No.	Pressure[1] (torr)	Volume % CH_4	CO	N_2	Light Hydrocarbons	O_2	Ar	CO_2	NO_x
17-03925	665	20	8	63	0.3	8	0.8	0.2	
17-03919	1396 (27psia)	85	10	4	0.1	---	0.2	---	
17-03933	1190	55	17	24	0.3	3.5	0.5	---	
17-03907	896	49	11	38	0.2	1	0.6	---	
17-03898	928	62	11	26	0.3	0.2	0.4	---	
17-03909	613	3	3	73	trace	19	0.9	0.2	
17-03920	878	51	11	31	0.2	6.0	0.5	---	
17-03899	730	51	14	26	0.2	7.5	0.5	0.9	trace
17-03905	828	22	11	62	0.4	3	0.9	---	
17-03914[2]	1000	18	5	62	0.3	13	0.8	0.2	
17-03927	650	41	14	43	0.4	0.7	0.7	---	
17-03906	1066	14	7	67	0.4	11	0.8	---	
17-03932[2]	612	3	2	78	0.1	16	1	---	trace
17-03911	618	48	10	33	0.2	8	0.5	---	trace

(1) atmospheric pressure on analysis date = 612 torr.

(2) high N_2 and O_2 values indicate possible leak when sampled.

Note: trace hydrogen chloride (HCl), Chlorethene ($C_2H_3Cl_3$), carbon tetrachloride (CCl_4) and hydrogen sulfide (H_2S) were noted in all 14 drums.

Appendix KK

Letter from E. S. Ryan to G. E. White

August 19, 1963

G. E. White

cc:
J. G. Epp
M. E. Maas
File (Record)

HISTORY REPORT - PROCESS WASTE DISPOSAL GROUP - JULY, 1963

1. Personnel: Salary 3; Hourly 6

2. Facilities: The repair of the defects in the filter bank and the relocation of the plenum in the Building 74 addition were started by the Sheet Metal Shop.

   The transfer of the liquid wastes in Pond No. 2A to Pond No. 2B was made by the Pipe Shop. A small heel of acid wastes remaining in Pond No. 2A was neutalized by pumping the basic liquid wastes in the south section of Pond 2B into Pond No. 2A. The liquid waste was then pumped from Pond 2A to the north section of Pond No. 2B.

   Men from the Service Group resumed drumming of the salts and sand in Pond No. 2A.

   A small scale experiment was made to test the burning capabilities of the lining of Pond No. 2A. The planking would not support combustion of its own and requires an additional fuel for rapid destruction.

3. Safety and Security: A safety and security meeting was held on July 29, 1963.

4. Trips: E. S. Ryan attended discussion meetings on July 8 and 9, 1963 at Oak Ridge National Laboratories, Oak Ridge, Tennessee. These discussions dealt with methods for disposal of contaminated liquid wastes by evaporation and deep well.

5. Visitors: Ray Miller, A.E.C., Health and Safety, ALO, Albuquerque, New Mexico, visited the Rocky Flats Division on July 25, and 26, 1963.

6. Operations: Table A is a summary of the liquid wastes, treated and untreated, which were released under the supervision of the Process Waste Disposal Group.

Table B lists the high and low of Ponds No. 1, No. 5 and No. 9.

Table C lists the drums of contaminated wastes in storage by the various buildings. Released drums of wastes were moved to Building 63 from the production areas.

Table D is a breakdown of 6 trailer shipments to Idaho Falls, Idaho.

Table E is a summary of the aqueous liquid wastes received, processed and released from Building 74.

The following radioassay results are on weekly composites made from daily samples of the effluent from the drainage tile.

Week of 7-1-63 - 7-5-63	200 d/m/l
Week of 7-8-63 - 7-12-63	150 d/m/l
Week of 7-15-63 - 7-19-63	170 d/m/l
Week of 7-22-63 - 7-26-63	150 d/m/l

Analysis of Well Waters Sampled July 25, 1963

Well	pH	d/m/l	NO_3 ppm	Sp Gr @ 27°C	Depth of Water 7-25-63	6-21-6
No. 1	7.6	40	280	1.003	7' 2"	7' 6"
No. 2	8.1	10	1,100	1.000	8' 7"	6' 9"
No. 3	8.1	20	440	1.001	15' 4"	12' 2"
No. 4	7.7	5	3,400	1.003	12' 4"	11' 6"
No. 6	8.1	15	10	1.000	3' 10"	5' 7"

The wells were not bailed during the month.

Well No. 5 remains dry.

No oils or still bottoms were burned during the month.

7. Future Problems: A new method for the sampling of the laundry waters released by Building 42 is needed. The present method of grab samples by the laundry operators, whenever other operations permit, does not supply a representative sample. A sampler controlled by the cycling mechanism of the washers would give a sample which would have more meaning.

The disposal of cyanide wastes which are produced in Building 44 are a potential problem. In the past, these wastes were set-up with Portland Cement in Building 44. At the present time, these wastes are being sent to Building 81 for destruction. The presence of flouride and Building 44 material in the waste makes this method undesirable for Building 81.

8. Development Work: F. E. Butler, Process Chem, CRDL, reports no work on Waste Disposal projects because of a rush project for Midland.

K. Fry, Analytical Labs, Building 71, began a study on the feasibility of concentrating radioactive material with the iron hydroxide floc. This study convers the repeated use of the floc in successive batch treatments of aqueous wastes. If successful, the process could lead to the recovery of the material from the iron floc.

E. S. Ryan

E. S. Ryan

ESR:bls

Appendix LL

Investigation CCl_4 Carbon Tetrachloride by R. W. Hawes

INVESTIGATION CCL4 CARBON TETRACHLORIDE

11-27-85 R.W.HAWES

CONTACTS PHIL SHOEMAKER Foundry 707 x-7959 page 4000-108
KIETH GROSSAINT Labs 559 x-2154
RICK GETTY Labs 559 x-4791
WENDY HENDERSON Manufacturing 707 x-4705
PAUL KING Foundry 707 page 4000-280
FARREL HOBBS 779 x-7431
ROCKY PETROCCHI Industrial Hygiene

Shoemaker confirmed the figures supplied by Setlock on the CCl4 usage. Hobbs figures usage at 14,000 gallons in the last 12 months

The other substance used to clean chips is Freon TF. Estimated at 55 gallons per month in B module, 55 gallons per month in C module, 25 to gallons per month in 776 and small amounts used in A module. This will combine with the carbon tet and be disposed of in the same facility. Estimated total usage 130 gallons per month. Hobbs says that the Freon is used for density determinations more than cleaning and is discarded frequently but the usage is probably right.

The evaporation rate of this Freon is about one third the rate of evaporation of carbon-tet.

Other substances used;

Texaco Regal cooling oil known as Regal R&O or Regal 645
Mobile 643 hydraulic oil
Tra-Con lubricating oils for the machines.
Total usage of these oils discarded to the same tank as the carbon-tet is about 28 gallons per month.

The waste tanks used are the pencil tank V-32 in the pit of module C, of building 707 and tank 1103 in building 1103. Currently tank V-32 empties into tank 1103 where it is sampled for plutonium before it is turned over to building 774 Liquid Waste Processing.

The primary usage of the carbon tetrachloride is to wash the oils off the metal chips. This is done prior to briquetting the chips for the foundry.

Paul King is collecting 500 ml samples from the waste tanks and will send them to 559 labs (11 a.m. 11-27-85).

TCA
Hobbs says that 1,1,1 Tri chloralethane is used in a closed cleaning system and only trace ammounts would be found in the waste liquids. Trade name "Chlorthene V6" usage estimated at 500 gallons per month. (W.A.G.)

Appendix MM

A Control Design for Plutonium Counting Systems

R01528URFP

A CONTROL DESIGN FOR PLUTONIUM COUNTING SYSTEMS. SPEECH.

by
L. W. Doher and J. D. McBride

For Presentation at the 11th Annual Meeting
of the
Institute of Nuclear Materials Management

Descriptors
Nuclear materials management
Plutonium
Radiometric analysis
Safeguard

May ~~24~~ 26, 1970
Gatlinburg, Tennessee

ABSTRACT

This paper describes a design which assures control of the plutonium counting systems used at RF to measure solid plutonium in waste and process materials, while establishing measurement bias and variability information for process control and inventory management.

INTRODUCTION

Chemical processing of plutonium offers many challenges because of this element's unique characteristics and because of the special administrative controls that surround it. The challenges include (1) the physical containment of plutonium for health reasons, (2) the danger of nuclear excursions, and (3) the accountability required of plutonium. This paper deals with the control of one type of measurement of plutonium for safeguard and accountability reasons. The method of measurement is radiometric, which is non-destructive.

At the 10th Annual Meeting of the Institute of Nuclear Materials Management, O. H. Willoughby and D. R. Cartwright of the Dow Rocky Flats Research and Development Department described the development of a non-destructive method for assaying plutonium in waste and process residue.[1] This method is radiometric and is classed as "passive"; that is, it uses the inherent radiation from the plutonium as a basis for measurement.

For a description of the measurement systems, two paragraphs from the Willoughby-Cartwright paper are quoted, as follows:

"The first radiometric system, the drum counter, was

Slide 1

installed in 1964. The drum counter geometry consists of an annular array of eight 30-inch halogen-quenched Geiger-Müeller detectors and 16 boron trifluoride neutron detectors within a shielded cavity. Fifty-five-gallon drums of contaminated waste are lowered into the cavity with a hydraulic hoist. Cadmium shielding is wrapped around

[1] RFP-1325, "Measurement of Plutonium in Process Materials and Contaminated Waste," O. H. Willoughby and D. R. Cartwright; a speech presented to the 10th Annual Meeting of the Institute of Nuclear Materials Management, April 28 - 30, 1969, at the Stardust Hotel, Las Vegas, Nevada.

the gamma detector to reduce the effects of low-energy radiation from americium on plutonium measurements. Electronic pulses resulting from radiation passing through the detectors are amplified, shaped, and scaled using standard nuclear instrumentation.

The early successes with the drum counter led to the development of the can counter, which is similar in design to the drum counter but smaller. The can counter performs plutonium

Slide 2

assays of gallon-size waste or residue packages in the process area where the wastes or residues are generated. The speed of the analysis and the easy acquisition of data have been of great benefit to production personnel in making accountability and management decisions for further waste and residue processing."

Existing data indicate that the can and barrel method of measurement is superior to the classic method of sampling and subsequent analysis for plutonium by laboratory methods, primarily because sampling errors are removed by measuring entire batches with the counting geometries.

The measurement systems described above provide the instrumentation necessary to detect plutonium, but the raw hardware by itself cannot assay the amount of plutonium present without the use of standards for comparison.

Consideration of standards for use with the geometries presents a two-fold problem: (1) the sample medium and the geometry affect the response of the detection systems, and (2) the amounts of plutonium required to provide multipoint, standard curves derived from individual standards are so great as to be economically impractical. Control of routine measurements at RF depends upon comparison with absolute standards, which must overcome the problems just mentioned.

In order to solve these problems, the concept of a modular standard was adopted in which each standard consists of modules appropriate to the geometry. For the drum counter, the modules are 1-gallon plastic bottles within a 55-gallon barrel; for the can counter, the modules are plastic-bagged material in a 1-gallon plastic bottle. Each module for each category of residue contains either (1) dry plutonium dioxide plus the average medium for that category, mixed until they are homogeneous, or (2) the medium only. Such a concept permits wide variability in both the amount of plutonium contained in the standard and the plutonium per unit weight.

The standard modules are prepared by mass measurement of Rocky Flats stream plutonium in synthetic, average media, rather than by sampling and destructive testing of actual residue.

Slides 3 and 4

The plutonium for these standards is processed as follows: (1) plutonium peroxide is received from Chemical Operations; (2) the plutonium peroxide is calcined to the stoichiometric ratio of plutonium dioxide ($PuO_{1.98}$) at 850° C. for 100 hours; (3) the oxide is then analyzed and the plutonium content corrected for metallic impurities, such as Am^{241}; and (4) isotopic ratios are determined by mass spectrometry, for calculating the atomic weight.

Both the media and the dry plutonium dioxide are measured and loaded into the modules so that the outer surfaces of the modules are free of alpha radioactivity. The modules can then be safely manipulated inside their 55-gallon and 1-gallon containers.

A standard for the drum counter consists of 27 wide-mouth plastic bottles. To simulate the heterogeneous characteristics of process plutonium residue in the barrels, some of these bottles are standard modules containing plutonium dioxide, some are empty bottles, and some are bottles containing residues free of radioactivity. (Slide 5 demonstrates the concept and versatility of the standard for the drum counter.)

Slide 5

A standard for the can counter consists of nine standard modules which are plastic bags that may either contain plutonium dioxide or may be "blank". Thus, a can standard possesses the same characteristics as a drum standard.

Slide 6

RF categorizes process residues according to their origin, and therefore their physical and chemical characteristics. Because the medium of these residues affects the radiometric response in the two counting systems, the Standards Laboratory is obliged to synthesize standards representing these same categories.

Standardization for category of residue requires multiple working standards that permit variations in standard values. This is possible, using a minimum amount of plutonium, because the working standards are made up of modular standards. With these standards, R and D personnel can provide standard curves for each category. The curves are normalized using both ideal (homogeneous) standards and non-ideal (heterogeneous) standards for each datum point. Therefore, a certain amount of variability is represented in the standard algebraic expressions because of the heterogeneous nature of RF solid residues.

The standards are also used in the RF measurement control program. Why a measurement control program?

The former RFD procedure for the measurement of amounts of nuclear material involved the following separate operations: (1) each batch of material was weighed or measured by volume, and sampled; and (2) the sample was analyzed for SS material content.

Because the analysis can never be exact, and because the sample cannot be considered truly representative, the measurement may be inaccurate. Therefore the quantities of plutonium used in computing a material balance are inexact. It becomes important, then, to determine just how inexact or biased the measurements are.

The RF measurement control program evaluates the systematic errors and their variabilities. Measurements of a given characteristic(plutonium assay) are compared with other measurements of the same characteristic made with an accepted standard. The second, or standard, measurement may be considered absolute because as a weight measurement it is vastly superior to the first, or observed (counting) measurement. The average difference between the observed and the standard values is the bias of the measurement. The standard deviation of the bias is a good measure of its variability.

The measurement control program is divided into categories of process materials and residues which are assayed for plutonium content in the counting geometries. Each program is assigned a Nuclear Materials Management (NMM) description code number. The Chemistry Standards Laboratory changes the values of the plutonium inside each standard by manipulating the modules, records these changes, and submits at least five such standards per month to be measured. The counting personnel measure the plutonium

Slides 7 and 8

in each standard and transmit the observed value to the Chemistry Standards group, which compares the values by subtracting the standard from the observed value. Thus, the validity of the measurement system for a particular residue category is established.

At the end of the month, the measurement control program numbers, the standard values, and the observed values are summarized by computer according to program and category as follows:

Slides 9, 10, and 11

(1) Each standard value is subtracted from its corresponding observed value; (2) an average of these differences is determined and (3) the standard deviation of the differences is calculated.

The average difference determines the mean bias per type of residue and the standard deviation around this average estimates the expected variability of each bias. The variability establishes realistic limits for a given bias, which in turn helps determine the disposition of residues, the validity of counter measurements, and the accuracy of inventories. The limits of error may then be calculated using these statistics.

In a recent six-month period, the measurement control program reflected biases from 1 percent to 20 percent and variabilities ranging from 4 percent to 37 percent. The measurement category showing the 37 percent variability was considered out-of-control and the production material surrounding the standard evaluation was remeasured following the appropriate remedial action.

CASE HISTORY

A portion of the RF plutonium chemical processing involves a purification process using ion exchange resin. The spent resin becomes a residue which is either leached to a discard level or incinerated for plutonium recovery. Disposition of this material must be decided after it has been packaged, removed from the dry-box system, and stored in 55-gallon drums. The spent resin is measured by the drum counter. The Standards Laboratory establishes a control for this drum counting measurement, as previously described.

During one period, the measurement control showed a bias as great as 37 percent. Such a bias in any measurement make it impossible to make a decision. If the bias is unknown, a decision could be made based upon an assay that is assumed to be valid and the decision would probably be wrong.

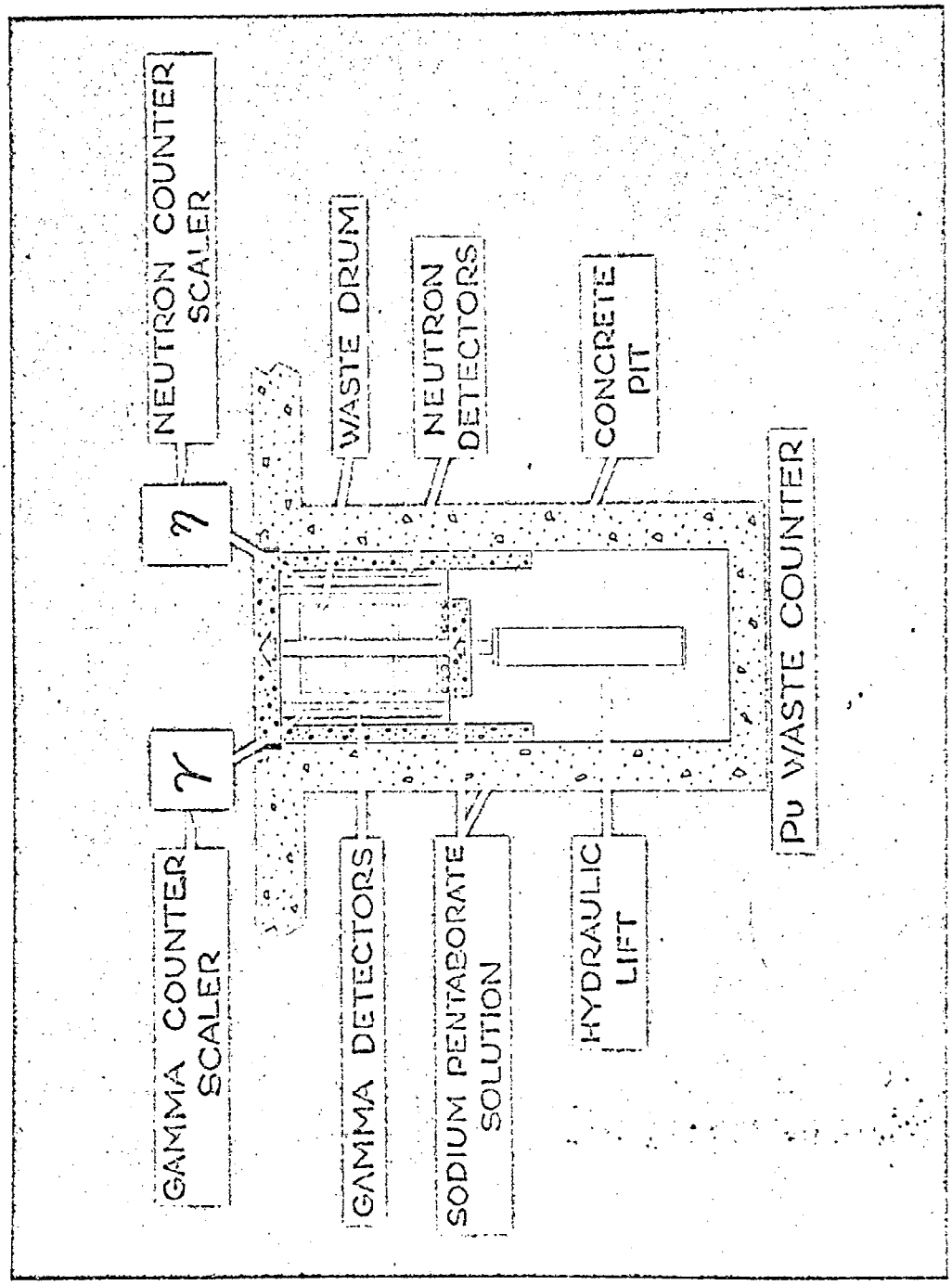
Fortunately, the bias was known from the measurement control program and remedial action was taken. All measurement was halted. The instrumentation and counting procedure were investigated and revised as necessary. All drums assayed from the last acceptable control count to the unacceptable one were recounted and the records adjusted. Disposition was made based on the recounted values. Control was re-established and measurements continued.

SUMMARY

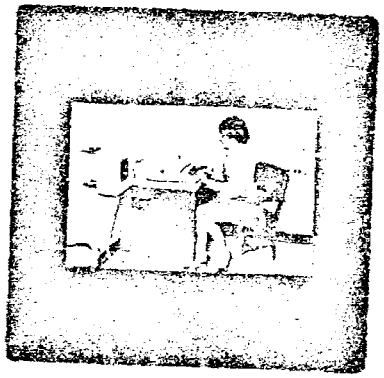
The Rocky Flats Division of The Dow Chemical Company assays plutonium in solid chemical process materials and contaminated wastes by non-destructive, radiometric techniques. Such working measurements were new to RF a few years ago and, therefore, the Chemistry Standards Laboratory was obliged to design a system that both standardized the instrumentation and placed measurement controls around it. Both calibration and working standards were designed and tested. These simulated those plutonium-bearing, solid, process materials and residues measured by the two types of counting geometries developed and currently in use at RFD. These standards have two constant external configurations, but they are highly variable in their internal configurations and their ranges of standard values. Thus, the standards are able to evaluate (1) the calibration curves, (2) the geometries and electronics, and (3) routine process measurements, under true working circumstances. The measurement control programs, which developed from the design, supply bias and variability information from (1) interplant shipments, (2) in-process items, and (3) discards. The controls are therefore useful management tools and proceed on a continuing basis.

ACKNOWLEDGEMENTS

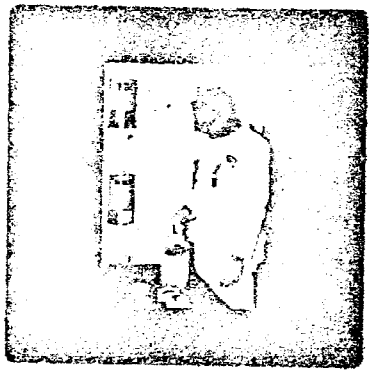
The authors wish to thank O. H. Willoughby and D. R. Cartwright for their technical assistance, W. R. Meininger and the Chemical Operations Staff for the process-related information, and the Technical Writing group which assisted in preparing this paper.



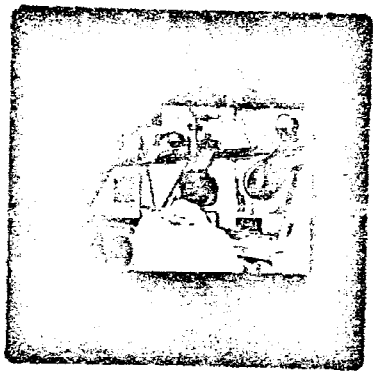
SLIDE 1: DRUM COUNTER SCHEMATIC



SLIDE 9

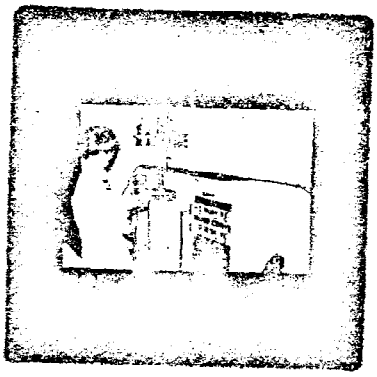


SLIDE 8

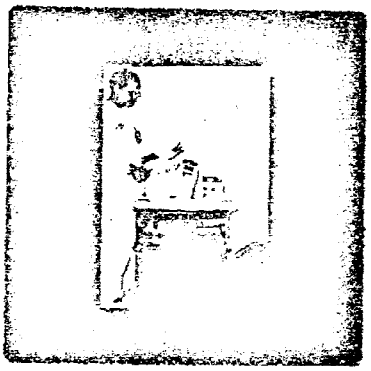


SLIDE 3

SLIDE 4



SLIDE 2



SLIDE 7

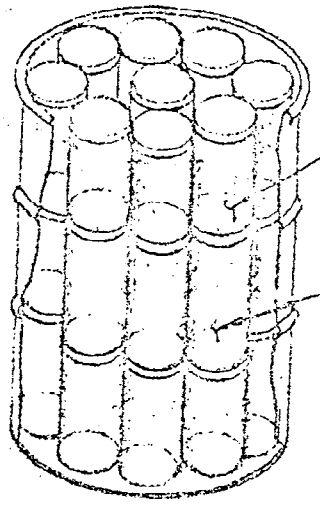


FIG.2. COMBUSTIBLES AND WASHABLES BARREL COUNTING STD.

SLIDE 25

DRAFT

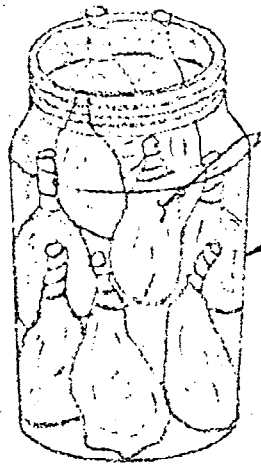


FIG. 3 TYPICAL CAN COUNTING STD.

SLIDE 6,.

STATISTICS - CURRENT MONTH

N	Bias	S.D.	Avg.S.V.
5	-3	+4	102

MEASUREMENT CONTROL REPORT

Prog. No.	Description	Range
C400	Contained Pu in Resin by Barrel Count	0 - 175 g

Appendix NN

A Plutonium Waste Counter

Trans. Am. Nucl. Soc. 13, *2, 748, (1970)
Appendix 12-17

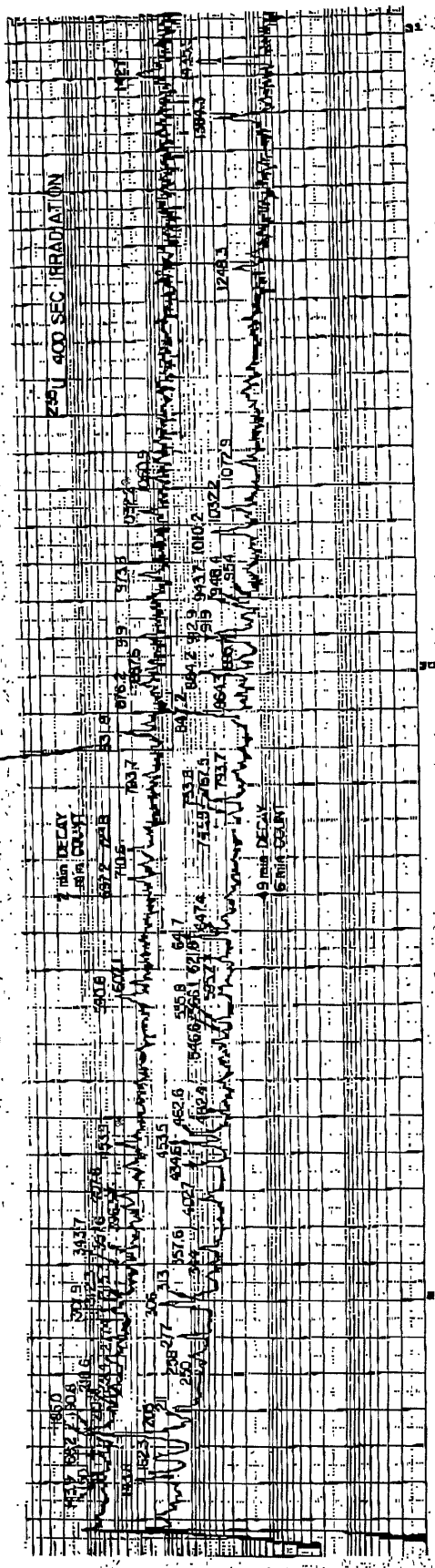


Fig. 1. Portions of fission product gamma-ray spectra following a 400-second bremsstrahlung irradiation of a ^{235}U sample. End-point energy of the bremsstrahlung beam 8 MeV.

line can be improved by optimizing the irradiation, decay, and count times. However, the complex gamma spectrum requires high resolution spectrometers and analysis procedures to accurately extract the gamma-line intensities.

An isotopic assay has been performed on some composite samples of uranium, thorium, and carbon. Using the 12 most prominent gamma lines, the amounts of ^{233}U and ^{232}Th were determined with an overall accuracy of better than 8%. The ^{235}U content was measured to be within 2% of the known amounts.

1. N. C. RASMUSSEN, J. A. SOVKA, and S. A. MAYMAN, "The Non-Destructive Measurement of Burnup by Gamma-Ray Spectroscopy," *Proc. Symp. Nuclear Materials Management*, pp. 829-48, IAEA (September 1965).

2. S. KALCOFF, "Fission-Product Yields from Neutron-Induced Fission," *Nucleonics*, 18, *11*, 201 (1960).

RFP-1584

4. A Plutonium Waste Counter,* *J. L. Lawless, R. N. Chanda (Dow)*

An operational system designed for the measurement of large quantities of plutonium contained in gallon-sized packages is described. The system performs a passive measurement employing both the 384-keV gamma-ray complex of ^{239}Pu and spontaneous fission neutrons from ^{240}Pu. Also included is a provision for measuring and correcting for the gamma-ray attenuation in the sample.

A block diagram of the system is shown in Fig. 1. Several unique construction features are contained in the design, including the selection and arrangement of shielding and the scanning mechanism. The sample rests upon a turntable which causes the sample to describe a helical

TABLE I

Evaluation of Helix Counter for Graphite and Ash Standards

Standard Value (grams)	Bias (%)	Precision (%)	Bias at 95% Confidence Level (%)	Precision at 95% Confidence Level (%)
Graphite				
10	6.3	1.1	8.5	2.3
25	9.8	1.2	12.3	2.6
50	5.1	1.0	7.1	2.1
100	2.9	0.8	4.5	1.7
200	-0.4	2.0	4.3	4.1
350	2.2	2.5	7.2	5.3
500	-2.7	5.8	14.2	12.1
Ash				
10	6.0	1.6	10.0	7.0
25	2.6	2.0	6.8	7.6
50	-2.0	1.4	3.4	5.2
100	-0.1	1.8	3.7	6.7
200	-0.5	1.6	3.8	8.0
350	2.3	4.4	11.1	16.4
500	-4.2	4.0	12.1	14.8
698.6	-4.1	3.6	12.1	14.2

*Sponsor: L. A. Matheson.

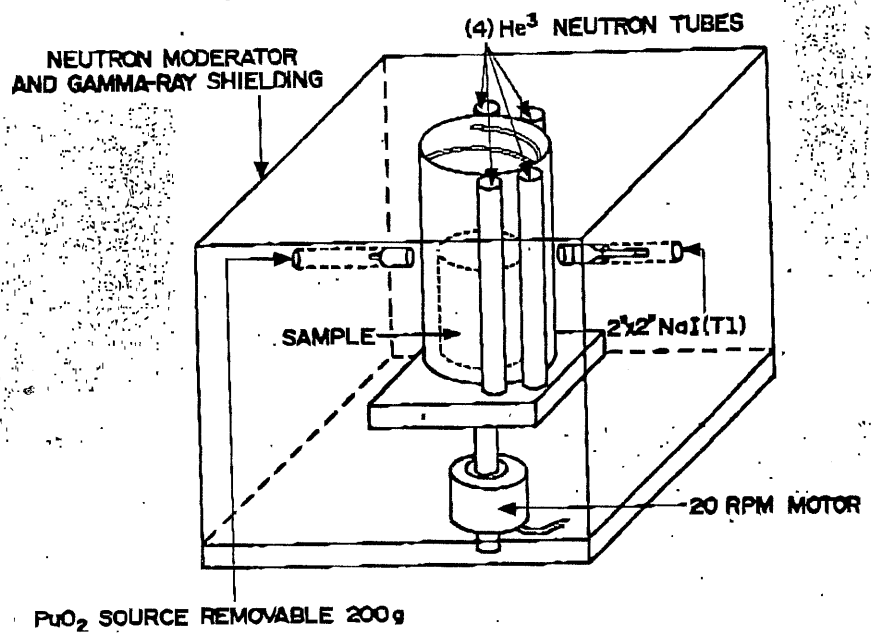


Fig. 1. Helix counter.

path between a 2- × 2-in. NaI(Tl) detector and a 200-g PuO_2 gamma-ray attenuation measurement source. Two 100-sec scans are performed, one of which is made with the external plutonium source for the sample attenuation measurement. The NaI(Tl) gamma-ray response is recorded on a single-channel analyzer set to effectively bracket the 384-keV complex. A simultaneous gross neutron count is performed using four ^3He neutron detectors. Conventional commercial electronics are used throughout. The entire system including electronics, moderator, and shielding rests upon a movable cart.

The precision and accuracy of the counter has been measured using certified standards of graphite and ash matrices.[1,2] Each standard was counted 5 times and the data subjected to statistical analysis. Over a range of 0 to 700 g of contained plutonium, a precision was obtained of $\leq 6\%$ with a bias of $\leq 10\%$. At the 95% confidence level, this corresponds to a bias of <14% with a precision of <16%. This system has been in operation at Rocky Flats for the past year.

1. J. L. LAWLESS, Y. M. FERRIS, and J. D. McBRIDE, "Evaluation of the Helix Counter Incinerator Ash Study," CRD-940232-104-C, Rocky Flats (April 1970).
2. J. L. LAWLESS, Y. M. FERRIS, and J. D. McBRIDE, "Evaluation of the Helix Counter Virgin Graphite Study," CRD-940232-104-D, Rocky Flats (April 1970).

5. Nondestructive Gamma Assay of Plutonium-Contaminated Solid Waste in Drums, *Alexander J. Dukat, John Gonser, Dean B. James (NUMEC)*

Nuclear materials management and safeguards as well as operations control require that drums of plutonium-contaminated wastes be assayed for their plutonium content. Destructive analytical techniques are not practical because of the difficulty of obtaining representative samples. A nondestructive analysis should provide a simple and rapid analysis of total plutonium content of the drum, with no bias resulting from physical and geometrical configurations within the drum.

The measurements were made with a 55-cm^3 Ge(Li) detector and a Nuclear Data 2200 series multichannel analyzer as described previously.[1] The drum was rotated at 4 rpm on a turntable while being counted by the stationary detector. A previously determined environmental background was subtracted, and an approximately 85-keV portion of the "384-keV" complex was integrated. A horizontal Compton correction was based on the activity in a 20-keV portion of the spectrum just higher in energy than the integrated portion of the spectrum. A correction was made for the self absorption of the solid material within the drum by comparing the activity due to a planar plutonium source shining through the unknown drum to that activity produced by the same source shining through a dummy drum.[1]

With an operationally reasonable scan time of 1000 sec of live time and a detector-face-to-barrel-center distance of 54 in., the calibration coefficient measured with a drum containing varying numbers of known plutonium packages with PVC-film matrices was determined to be 1.1019 (g sec)/count.

Using twice the standard deviation of the background as a criterion for detection, 0.07 g of ^{239}Pu may be determined. Experimental one-sigma precision for 1, 10, and 50 g of ^{239}Pu is ±0.12, ±0.33, and ±0.87 g, respectively, as determined by repetitive measurements.

The extent of the loss in accuracy with package location in the drum was determined by filling a drum with three layers of seven, 1.5-gal cardboard packages each, all of the same matrix material, and then alternately substituting one package with a plutonium-containing package. With reference to a drum containing plutonium uniformly distributed throughout, a package in the center of the drum showed about an 11% reduction in activity; packages on the bottom-layer edge showed a 3% reduction in activity; while packages on the top layer showed a 6% increase in activity.

The necessity of segregating materials into drums with similar gamma absorption density was demonstrated. Packages of real waste were assayed by the package-assay system[1] and randomly placed in drums without regard to plutonium content or self-adsorption correction.